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Multi-Impact Durability and Processing of Thick-Section Carbon-Glass/Epoxy Hybrid Composites Toughened With Thermoplastic Polyurethane Inter-Layer Films

by Steven E. Boyd and James P. Wolbert

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14. ABSTRACT

Research was conducted on 3-D (three-dimensional) carbon and glass hybrid epoxy composites toughened with thermoplastic polyurethane (TPU) film inter-layers. The study focused on the effect of sample processing methods on impact degradation rates. The newly developed low velocity impact, four quadrant multi-hit testing protocol described in other works by Emerson et. al (2010 and 2011) and Boyd et. al (2011), which are referenced in this report, and has been successfully used to rank thick-section (>13 mm) composite materials and is used here. Samples were successfully processed using room temperature, standard molding techniques, and post-cures for the toughened SC-15 epoxy with little to no warpage and defects. The use of thicker sub-layers and strengthening of the composite interfaces with TPU inter-layers arrested damage progression, reduced overall delamination, and lowered stiffness degradation per impact. However, a mismatch in carbon and glass fiber properties in the hybrid samples produced extensive localized damage per impact and had the net effect of reducing sample stiffness.

15. SUBJECT TERMS

damage tolerance, hybrids, low velocity impact, processing parameters, thermoplastic polyurethane

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1. Introduction

The Army is currently investigating novel materials to replace tradition monolithic materials in Army structural applications. The leading composite material in these investigations is a two-phase toughened epoxy SC-15 reinforced with a woven glass fabric (*1*, *2*). Previous reports on the characterization of this composite have demonstrated that delamination is a critically important damage mechanism in low to medium velocity impacts (*3–5*, *6–10*). Strategies to mitigate the delamination and stiffness degradation in impacted composites include reducing the number of resin-rich interfaces by using thicker (>2–3 mm) sub-layers of reinforcement and toughening the remaining interfaces with thin thermoplastic polyurethane (TPU) films. The sub-layers may consist of 3-D (three-dimensional) pre-forms of a single fiber or two distinct fiber types. Hybrid materials, consisting of two different fiber types, offer a novel material solution to synergistically improve net mechanical properties of a composite material tailored to a particular application. This research focuses on carbon/glass fiber hybrid materials, which employee thicker sub-layers and interfaces strengthened with thin TPU films.

Since the hybrid composites contain two constituent fibers, processing the samples presents some unique challenges and provides motivation to experiment with different processing and curing methods. Carbon fibers compared to glass fibers have a negative coefficient of thermal expansion (CTE), higher stiffness, and lower fracture strain. Combining these two disparate fibers into a heterogeneous reinforcement for a composite material, if not performed with a proper cure cycle, may cause a warped and unusable part. Care must be taken to optimize the cure/post-cure of the samples to ensure a minimum of warpage and residual thermal stresses.

Inserting TPU film into the stacking sequence of the composite also presents its own processing challenges. TPU films are impermeable to resin and therefore restrict resin flow. If the infusion is not well controlled, defects may form through "race-tracking" of the infusion front. Another issue involves whether the TPU films are *placed* or *pressed*. A placed TPU film is inserted into the stacking sequence and cured in situ with the cure of the resin system used. A pressed TPU film is pressed into surrounding dry fiber sub-layers under pressure and heat. Once the stacking assembly is cooled, resin infusion is performed separately. Research conducted both at the U.S. Army Research Laboratory (ARL) and the Center for Composite Materials (CCM) at the University of Delaware indicates that placing or pressing the TPU film toughening inter-layers has a measurable effect of the composite properties.

The present investigation is ancillary to ongoing research in composite material durability at ARL. Hybrid materials consisting of carbon and glass with toughened TPU film interfaces are of special interest because of the potential to improve flexural stiffness and reduce sample degradation per impact. This materials-by-design approach will produce a more damage tolerant

and durable material for low to medium energy impact. Processing and curing methods for manufacturing hybrid materials present unique challenges and may influence mechanical and/or impact properties.

Five novel hybrid samples are processed with TPU film inter-layers and evaluated with the four quadrant, low velocity impact (LVI) protocol (*3–5*). High-fidelity measurements of the force-time history are recorded during each impact and are used to calculate deflection and effective sample stiffness. The degradation of sample stiffness during successive neighboring impacts is of key interest to measuring the damage tolerance of an Army-relevant material. Other complementary methods that provide insight to the mode of damage under such impact include ultrasonic measurements of delamination and visual inspection of damaged samples

2. Materials and Processing

2.1 Material Constituents

Two 3-D fiber architectures are utilized in the samples and one type of TPU film. The 3-D orthogonal, interlock architecture carbon pre-form was manufactured by Albany Engineered Composites (*11*). The pre-form consist of non-crimp IM7 carbon fibers and S2-glass stitching with an areal density of 4270 kg/m^2 (101.2 oz/yd^2). The 3-D orthogonal, interlock architecture E-glass pre-form was manufactured by 3Tex, Inc. (*12*). The pre-form consist of non-crimp E-glass fibers and stitching with an areal density of 4080 kg/m^2 (96.6 oz/yd^2). The TPU film used is Dureflex A4700 by Deerfield Urethane (*13*). It is a polyether based, aliphatic thermoplastic polyurethane film used here in 0.38 mm (0.015 in) thickness.

2.2 Standard Vacuum Assisted Resin Transfer Molding (VARTM) with SC-15 Resin

A standard vacuum assisted resin transfer infusion is performed at room temperature conditions. The composite part is stacked on a flat tooled surface, typically an inert sheet of glass or aluminum caul plate prepared with a release agent. The part is bagged twice; the inner bag for the resin infusion and the outer bag is a leak proof barrier, which maintains vacuum pressure after infusion. The part is debulked overnight with a vacuum pressure of at least 29 in of Hg (98 kPa). The part is infused with Applied Poleramic SC-15 resin (a two phase toughened epoxy) that is mixed from two parts (parts A and B) using the manufacturer's recommended mix ratios (*14*). Once infused, the part is cured using the standard SC-15 cure/post-cure cycle. The part is held at 95 °F for 1 h and then slowly ramped 0.5 °F/min to 140 °F until the resin has fully gelled in the part (this takes ~2 h). Once gelled, the composite part is post-cured by ramping at 4–5 °F/min to 250 °F and held for 3 h and then allowed to cool under vacuum overnight.

2.3 Processing of Hybrid Samples

All samples processed were relatively flat without measurable warpage. Due to a limited supply of carbon pre-forms, only one impact sample was manufactured for each processing trial (see table 1). Hybrids nos. 1, 3, and 5 were processed using the standard VARTM and SC-15 cure/post-cure. Hybrid no. 3 is the baseline material without TPU inter-layers.

Hybrid no. 2 is referred to as a "pre-pressed" sample in which the stacking sequence before infusion was pressed at 3 U.S. tons (115 kPa) for 15 min at 250 °F and then cooled. After cooling, Hybrid no. 2 was infused using standard VARTM and SC-15 cure/post-cure procedure. Hybrid no. 4 was infused under pressure in a 400-ton heated press. Know as "press-aided VARTM" (PA-VARTM) the procedure is usually used to produce composite parts with higher fiber contents per volume (*15*). The stacking sequence is single bagged and placed in the 400-ton press under vacuum. The temperature is raised to 120 °F (via the heated platens) and the bagged part held under 6 U.S. tons (230 kPa) of pressure. SC-15 resin heated to 120 °F is infused and monitored until gelled. The post-cure consist of raising the platen temperature to 140 °F for 1 h and then to 250 °F for 3 h. The gelled part is then slowly cooled under pressure to 90 °F.

2.4 Impact Samples

After the five successful samples were processed and post-cured, the samples were debagged and cut using a waterjet to a square 40.6- $\times$ 40.6-cm impact sample. Hybrid no. 4, the PA-VARTM sample, had a significant top-surface indent due to the distribution media used to encourage resin flow. The two "pressed" samples (see figure 15 in section 4.3) Hybrids nos. 2 and 4, had inclusions measuring 25.8 cm^2 (4 in^2) and 42 cm^2 (6.5 in^2), respectively.

Table 1. Hybrid samples tested for impact properties.

Composite (SC-15 resin)	Configuration and Lay-up (Stacked)		Processing	Thickness (mm)	Areal Density (kg/m²)
Hybrid No. 1		2 × 3-D IM7-carbon + 4 × 0.015 in TPU inter-layers + 3 × 3-D 96 oz E-glass	Standard VARTM	16.87	26.4
Hybrid No. 2 ("Pre-Pressed")		2 × 3-D IM7-carbon + 4 × 0.015 in TPU inter-layers + 3 × 3-D 96 oz E-glass	Pre-pressed TPU film /standard VARTM	15.52	25.0
Hybrid No. 3 (Baseline)		2 × 3-D IM7-carbon + 3 × 3-D 96 oz E-glass (no TPU)	Standard VARTM – NO TPU	14.66	24.1
Hybrid No. 4 (PA-VARTM)		2 × 3-D IM7-carbon + 4 × 0.015 in TPU inter-layers + 3 × 3-D 96 oz E-glass	PA-VARTM	14.91	24.7
Hybrid No. 5		2 × 3-D IM7-carbon + 4 × 0.015 in TPU inter-layers + 3 × 3-D 96 oz E-glass	Standard VARTM	16.15	26.0

3. Experimental Impact Testing

3.1   Drop Tower

An Instron Dynatup model 8110 impact test system was used for these experiments. The impactor was a 50-cm hemispherical tup and the strain-gage based load cell had a capacity of 222.4 kN (50 kips). Force data during each impact were recorded for 25 ms duration at a sampling rate of 270 kHz. Tup displacement was calculated by converting the force to tup acceleration and integrating twice with respect to time.

3.2   Impact Table

The impact table fixture used in the impact testing is shown in figure 1. The all-steel fixture consists of a 3.8-cm thick, 50.8-cm square table, bolted to a 2.5-cm thick base plate with four 5.1-cm diameter, 30.5-cm long legs. The top plate is 2.5-cm thick held in place with sixteen 2.2-cm diameter high-strength steel bolts. Figure 1 illustrates the simply supported condition with the addition of the adapter plate (designed to snuggly fit into the inner opening of the square table), four 1.3-cm steel rod supports that rest in machined "v"-grooves in the adapter plate, and a 0.3-cm thick 2.5-cm wide strip of soft rubber resting between the sample and top steel plate. The entire assembly is secured with sixteen hand tightened bolts to minimize rigid-body motion of the sample after impact and rebound.
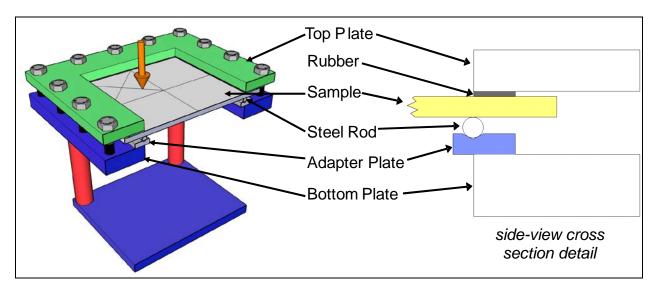

Figure 1. Exploded view illustration of the impact table, with detail showing a side-view cross-section of one side of the simple support.

3.3 Impact Location and Energy

Figure 2 illustrates the locations of impact for all samples in this investigation. Impacts were conducted one-at-a-time to permit non-destructive evaluation of each panel after each impact. Mass and height of the impactor were 227.4 kg and 96 cm, respectively, yielding an impact energy of 2141 J and velocity of 4.34 m/s.
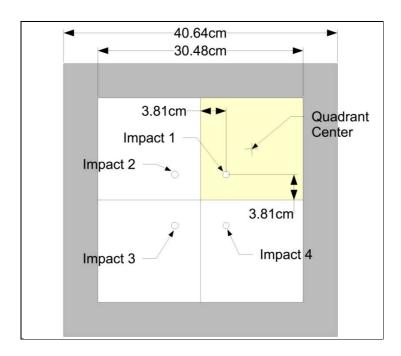

Figure 2. Illustration of the sample and impact location.

3.4 Non-Destructive Evaluation

The samples were removed from the impact table and ultrasonically scanned (producing a c-scan image) before and after each impact with a transducer frequency of 0.5 MHz and x-y resolution of 1 mm. These scans were used to track the evolution of damage in the sample. Delamination was observed as the predominant damage mode in the samples. All scans reported in this work were performed with the scanner operating in through-transmission mode. This approach provided an accurate determination of the extent of delamination across the full dimension of the samples.

4. Results and Discussion

4.1 Force vs. Displacement

Force-deflection curves are given for all hybrid samples in figures 3–7. The curves are generally separated into three regions: (1) the impact portion, (2) a transient phase involving damage formation and energy absorption up to the maximum sample deflection, and (3) the rebound with a "recovery" to a permanent sample deflection. The baseline Hybrid no. 3 (no TPU) shows the highest loss in stiffness per impact and maximum deflection (see figure 5) of any hybrid. These two results are due to the lack of a TPU film toughening inter-layer, which would improve the samples damage tolerance. One interesting trend displayed by these force-deflection curves is the extensive damage and impact energy absorption that occurs. Each hybrid displays a significant sample deflection between 2 and 3.5 cm. This is unique to the hybrids of this investigation and is not observed in similar areal density reinforced samples tested in prior studies (*3–5*). Two possible causes of this extensive damage include the mechanical properties of the E-glass used in the core and the disparate properties in the carbon and glass pre-forms. The impact energy of 2.1 kJ was originally selected for S2-glass composites (*3*). E-glass fiber composites have lower strength and stiffness than corresponding S2-glass composites and therefore experience more damage and deflection subject to the same impact energy. Also, the carbon pre-form is much stiffer than the E-glass core and produces large interfacial shearing stresses under impact, which cause TPU film inter-layer failure (see figures 15–19 in section 4.3). In summary, the hybrids with TPU film inter-layers perform slightly better than the baseline having slightly higher peak loads, less damage, and less deflection after four impacts.
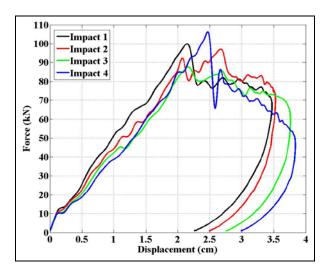

Figure 3. Force vs. displacement for Hybrid no. 1.
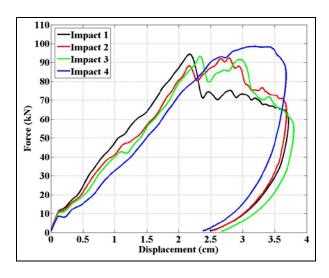
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Figure 4. Force vs. displacement for Hybrid no. 2
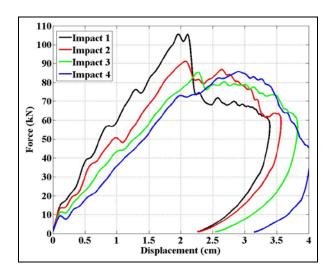("pre-pressed").

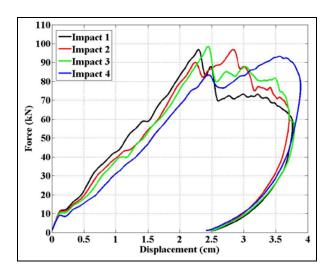Figure 5. Force vs. displacement for Hybrid no. 3
(baseline).

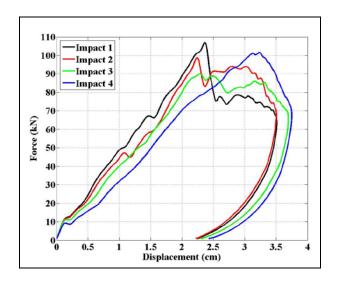Figure 6. Force vs. displacement for Hybrid no. 4
(PA-VARTM).

Figure 7. Force vs. displacement for Hybrid no. 5.

4.2   Damage Evolution – Ultrasonic Scans

Figure 8 gives ultrasonic c-scan images for all hybrids prior to impact testing. Pre-impact scans are performed for all samples tested and are critically important for revealing any processing issues that may compromise impact performance. Before discussing the results, it is important to understand that through-transmission scans identify inclusions and their extent but not their through-thickness location. As expected the baseline hybrid with no TPU film inter-layers had no initial defects and was processed successfully. Samples with TPU film had defects related to the impermeability of the TPU film layers, mostly dry fiber areas caused by "race-tracking" during infusion. Hybrid nos. 1 and 5, processed using standard VARTM, had some defects, but since the defects were located near the edges, the samples were judged to be successfully processed. The two hybrids that included a heat and pressure component in the processing phase had two significant inclusions. Although the inclusions can be located anywhere in the thickness, they are most likely at the interface between the pre-forms and TPU films. In both Hybrid nos. 2 and 4, the processing under heat and pressure of the stacked layers probably pressed the TPU film into surrounding fibers saturating them and inhibiting resin infusion. Regardless of the presence of the inclusions, the samples were tested for impact properties.
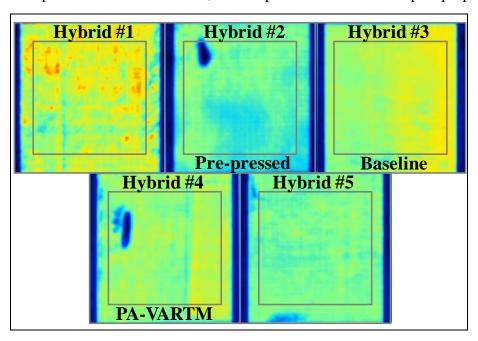

Figure 8. C-scan images showing initial undamaged state for all hybrids as labeled.

Figures 9–13 show ultrasonic c-scan images of damage progression for all hybrid materials. The best performer was Hybrid no. 1 (figure 9) with the lowest damage area progression per impact and damage contained largely in the vicinity of the hemispherical impactor. The baseline Hybrid no. 3 (figure 11) had the most damage area progression of any of the hybrids due to its lack of TPU toughening inter-layers. The two pressed hybrids, Hybrid nos. 2 and 4, had the most "fringe" damage showing up as lighter damage areas surrounding the main point of impact.

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These "fringe" areas are most likely delaminations of the interfaces, perhaps failure in the TPU film inter-layers. It is interesting to note that during successive impacts, the inclusions of Hybrid nos.2 and 4 do not appear to affect the impact damage or damage progression in the sample. The damage formation and progression of Hybrid no. 5 is similarly unaffected by edge defects; however, Hybrid no. 5 performs slightly worse than identically processed Hybrid no. 1.
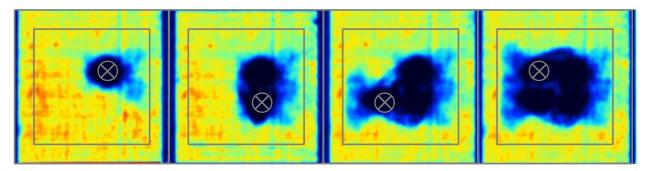

Figure 9. C-scan image showing progression of delamination in Hybrid no. 1.
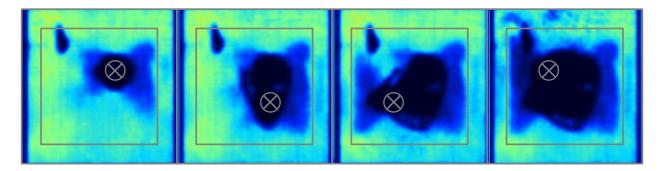

Figure 10. C-scan image showing progression of delamination in Hybrid no. 2 ("pre-pressed").
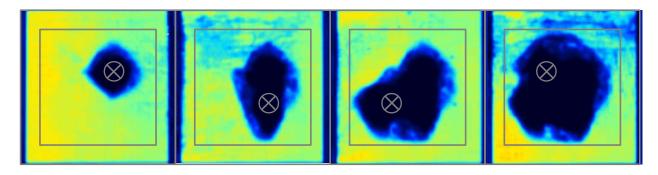

Figure 11. C-scan image showing progression of delamination in Hybrid no. 3 (baseline).
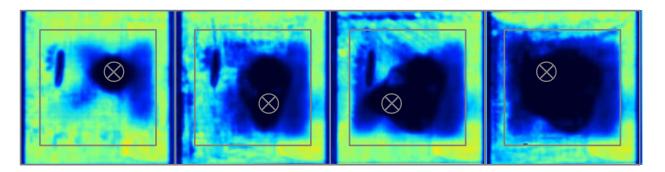
Figure 12. C-scan image showing progression of delamination in Hybrid no. 4 (PA-VARTM).
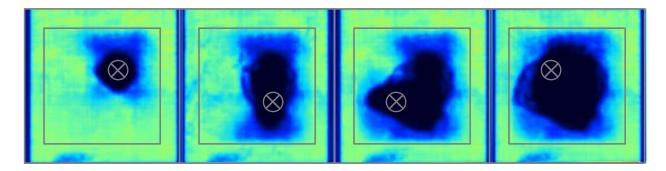
Figure 13. C-scan image showing progression of delamination in Hybrid no. 5.

Delamination area and progression in all hybrid samples is given in figure 14. The damage is presented as a percentage of aperture—area of the sample not constrained by the simply-supported boundary conditions (see figures 2 and 3). The key result here is that TPU film inter-layers significantly reduce and arrest the progression of impact damage. Without TPU inter-layers, there is a significant increase in damage after each impact. Hybrid nos. 1 and 2 performed best under impact as can be seen from the ultrasonic c-scans (see figures 9 and 10).

Figure 14. Delamination areas as a percentage of impact table aperture for each material.

4.3 Failure Mechanisms–Visual Inspection

Images of front and back face impact damage for each sample are shown in figures 15–19. From the force-deflection curves o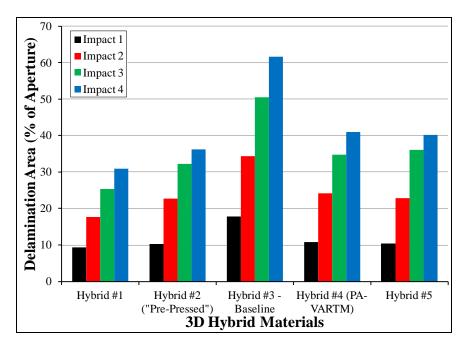f each sample (see figures 3–7); a significant amount of damage and energy absorption was indicated during impact. The images of figures 15–19 demonstrate that each sample experienced much localized impact damage directly under the hemispherical impactor and back face perforation resulting in a rectangular tab of the outer carbon pre-form separating from the hybrid assembly. The breakage is most severe in the area of the first and second impact indicating that the first couple of impacts caused more localized damage and punching shear perforation through the sample. After successive impacts, the failure transitions from localized shear to wide spread delamination as the damaged samples become more compliant to impact. From figure 17 for the baseline Hybrid no. 3, the damage is more severe revealing significant punching on the front face and perforation on the back face. From this visual evidence, the TPU film inter-layers provide a much improved damage tolerance and durability to the hybrid samples. However, severe damage observed on the back face images of figures 15–19 (b) suggested that a mismatch in carbon and glass properties may be causing a failure in the TPU inter-layers in the vicinity of the impact.
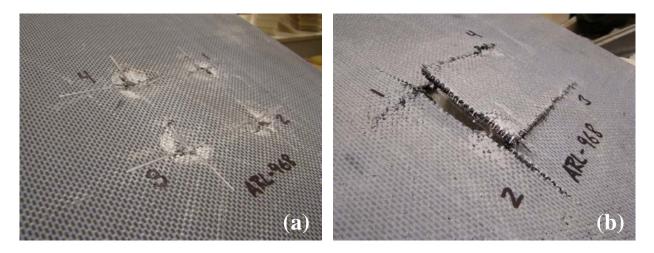
Figure 15. Front face (a) and back face (b) impact damage for Hybrid no. 1.
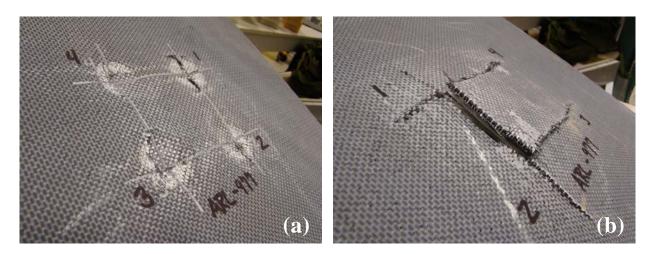

Figure 16. Front face (a) and back face (b) impact damage for Hybrid no. 2 ("pre-pressed").
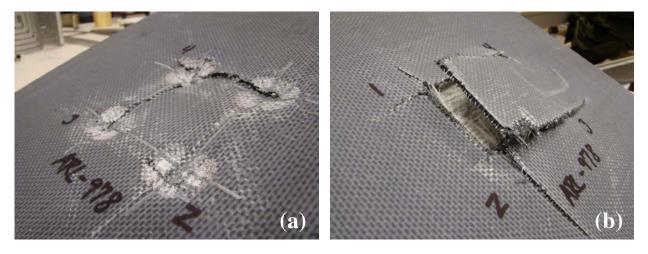

Figure 17. Front face (a) and back face (b) impact damage for Hybrid no. 3 (baseline).
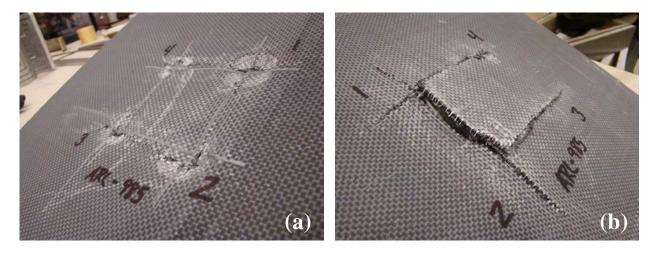
Figure 18. Front face (a) and back face (b) impact damage for Hybrid no. 4 (PA-VARTM).
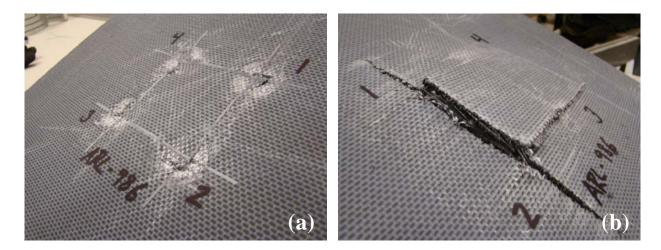
Figure 19. Front face (a) and back face (b) impact damage for Hybrid no. 5.

4.4   Degradation Rate

In the data reduction presented in (*4–5*) the extent of damage within the material is assumed related to the sample stiffness (slope of the force-deflection curve) during impact. For purposes of quantification the stiffness at 1 cm deflection is considered because the force-deflection curves (see figures 3–7) are still nominally linear up to that point and most of the transient vibration has dampened. The degradation rate (d_s) is defined as the slope of the linear fit to the initial stiffness versus impact, with units of kN/cm·impact.

The impact stiffness for all hybrid materials is listed in figure 20. The stiffest sample is the baseline Hybrid no. 3 without TPU film inter-layers. This result is surprising considering that Hybrid no. 3 had the most damage progression (see figure 14) and sustained the highest localized damage and energy absorption (see both figures 5 and 17). Considering that TPU films may be modeled as materials with hyper-elastic constitutive properties (*16*), the addition of 0.015-in

TPU film inter-layers may have reduced the flexural stiffness of the other hybrid samples versus the baseline sample. The observed decrease in sample stiffness from the baseline (Hybrid no. 3) to other identically processed hybrids (Hybrid nos. 1 and 5) containing TPU was not demonstrated in similar research involving S2-glass composites (*17*). However, as seen in both figure 20 and table 2, the baseline hybrid still had the greatest degradation rate per impact. This indicates that even though Hybrid no. 3 is a stiff sample, after two impacts, its stiffness reduction matches or exceeds that of the other TPU toughened hybrids.
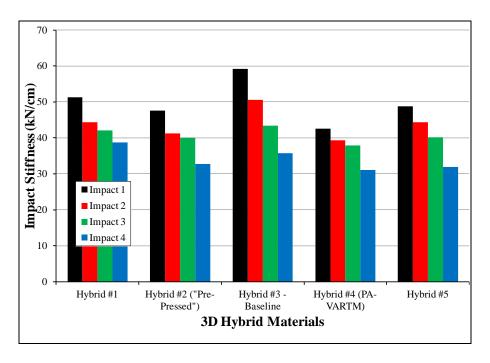
Figure 20. Initial impact stiffness for all hybrid materials.

Table 2. Degradation rates for all hybrid materials.

Material	Degradation Rate (kN/(cm*impact))
Hybrid no. 1	4.0
Hybrid no. 2 ("pre-pressed")	4.6
Hybrid no. 3 – baseline	7.7
Hybrid no. 4 (PA-VARTM)	3.6
Hybrid no. 5	5.5

Overall, the hybrid samples with TPU film inter-layers perform comparably. The worst performer is Hybrid no. 4 (PA-VARTM) having the lowest stiffness per impact of any of the TPU toughened hybrids. Important to note here is that both Hybrid nos. 2 and 4, which involved pressure in their processing steps, performed worse than Hybrid nos. 1 and 5, which were processed using standard VARTM. Although Hybrid nos. 2 and 4 had inclusions, it is difficult

to know the extent to which this influenced their impact performance. Hybrid nos. 1 and 5 performed well; however, Hybrid no. 5 sustained some increased damage during the fourth impact causing it to exhibit a higher degradation rate as list in table 2.

5. Conclusions

The processing of carbon/glass hybrid composites toughened with TPU inter-layers and its affect on impact properties were investigated. Results indicated that TPU toughened hybrid materials with thick-sections (>13 mm) can be successfully processed using standard VARTM and cure/post-cure for SC-15 toughened epoxy with little or no sample warpage and defects. The resulting novel materials perform well in impact testing evaluated using the newly developed, four quadrant, LVI protocol. The addition of a 0.015-in TPU film inter-layer at resin-rich interfaces significantly reduced delamination and damage progression under impact resulting in a more damage tolerant material. However, the TPU film inter-layers did reduce sample stiffness under impact resulting in slightly less durable composites. An excessive amount of localized impact damage and failure of adjacent TPU film inter-layers was observed in all hybrid materials due to the property mismatch between the carbon and glass reinforcing sub-layers.

The present ancillary investigation demonstrates much promise for the design of durable novel materials; however, much work is still to be done in the following areas. A modeling framework must be developed to fully investigate the strengthening mechanisms discovered empirically through using TPU film inter-layers in composite materials. This model must also be able to implement a material-by-design approach for the design of more durable and damage tolerant novel materials subject to low and medium energy impact. Many types of TPU films are commercially available and their unique properties need to be examined to find an optimal configuration (both TPU type and inter-layer thickness). Based on the results of the present investigation, in order to reduce the detrimental effect of property mismatches between disparate fibers, hybridized sub-layers should contain a balanced reinforcement of both fiber types. Such efforts are already underway at both ARL and the CCM at the University of Delaware.

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List of Symbols, Abbreviations, and Acronyms

3-D	three-dimensional
ARL	U.S. Army Research Laboratory
CCM	Center for Composite Materials
CTE	coefficient of thermal expansion
d_s	degradation rate
LVI	low velocity impact
PA-VARTM	press-aided VARTM
TPU	thermoplastic polyurethane
VARTM	Vacuum Assisted Resin Transfer Molding

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         J GARDNER
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         M VANLANDINGHAM
         B LOVE
         T BOGETTI
         S BOYD